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DEMANDE INTERNATIONALE PUBLIEE EN VERTU DU TRAITE DE COOPERATION EN MATIERE DE BREVETS (PCT)

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(71) Déposant (pour tous les Etats désignés sauf US): ROUSSEL-UCLAF [FR/FR]; 35, boulevard des Invalides, F-75007 Paris (FR).

(72) Inventeurs; et

(75) Inventeurs/Déposants (US seulement): MEINARD, Colette [FR/FR]; 24, avenue Petite-Suisse, F-13012 Marseille (FR). SUGLIA, Jean-Claude [FR/FR]; "Le Deven" n° 13, F-13112 La Destrousse (FR). TARANTA, Claude [FR/FR]; La Parade, Bâtiment Camus, F-13090 Aix-en-Provence (FR).

(74) Mandataire: TONNELLIER, Marie-José; Roussel-Uclaf, 111, route de Noisy, B.P. 9, F-93230 Romainville (FR).

(81) Etats désignés: AT (brevet européen), AU, BE (brevet européen), BR, CH (brevet européen), DE (brevet européen), DK (brevet européen), ES (brevet européen), FR (brevet européen), GB (brevet européen), IT (brevet européen), JP, KR, LU (brevet européen), NL (brevet européen), SE (brevet européen), US.

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Avec rapport de recherche internationale. Avec revendications modifiées.

(54) Title: NEW CONCENTRATED AQUEOUS EMULSIONS, PROCESS FOR PREPARING THEM AND THEIR USE IN THE FIELD OF PESTICIDES

(54) Titre: NOUVELLES EMULSIONS AQUEUSES CONCENTREES, LEUR PROCEDE DE PREPARATION ET LEUR APPLICATION DANS LE DOMAINE PESTICIDE

(57) Abstract

Concentrated aqueous emulsions, characterized in that they contain, in addition to the usual constituents of aqueous emulsions and the active principle, a film-forming agent and a surface-active agent chosen from the group of acid phosphate ethers. The emulsions are used, in particular, in the field of pesticides.

(57) Abrégé

L'invention a pour objet des émulsions aqueuses concentrées caractérisées en ce qu'elles renferment en plus des constituants habituels des émulsions aqueuses et du principe actif, un agent filmogène et un agent tensio-actif choisi dans le groupe des éthers phosphates acides. Les émulsions de l'invention trouvent notamment leur application dans le domaine des pesticides. CALLINAN LAWRIE 278 High Street, Kew Victoria 3101, Australia Facsimile (613) 853.0062

NOTICE OF ENTITLEMENT

We, ROUSSEL-UCLAF of 35, Boulevard des Invalides, F-75007, Paris, France being the applicant and the person nominated for grant of patent in respect of Application for an invention entitled "NEW CONCENTRATED AQUEOUS EMULSIONS, PROCESS FOR PREPARING THEM AND THEIR USE IN THE FIELD OF PESTICIDES" state the following:-

The actual inventors of the invention are as follows: MEINARD, Colette; 24, avenue Petite-Suisse, F-13012 Marseille, France; SUGLIA, Jean-Claude; "Le Deven" n° 13, F-13112 La Destrousse, France and TARANTA, Claude; La Parade, Bâtiment Camus, F-13090 Aix-en-Provence, France.

The basic application(s) FR 89/02 092 filed 17 February 1989 is the first application(s) made in a Convention country in respect of the invention.

The person nominated for the grant of the patent is the applicant of said basic application and would, if a patent were to be granted upon an application made by the said actual inventors, be entitled to have the patent assigned to it.

8ignature

Jeffrey A. Ryder

Patent Attorney for the Applicant

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(54) Title
NEW CONCENTRATED AQUEOUS EMULSIONS, PROCESS FOR PREPARING THEM AND THEIR USE
IN THE FIELD OF PESTICIDES

International Patent Classification(s)

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(71) Applicant(s) ROUSSEL-UCLAF

(72) Inventor(s)

COLETTE MEINARD; JEAN-CLAUDE SUGLIA; CLAUDE TARANTA

(74) Attorney or Agent CALLINAN LAWRIE , Private Bag 7, KEW VIC 3101

(56) Prior Art Documents
AU 566470 24492/84 AO[1N25/04
AU 597314 65821/85 AO1N25/30

(57) Claim

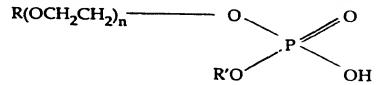
- 1. Concentrated aqueous emulsions comprising:
 - 0.01 g to 250 g/l of pyrethrinoid;
 - g to 350 g/l of aromatic hydrocarbons;
 - 0 g to 350 g/l of phthalic ester;
 - 40 g to 200 g/l of acid phosphate ether;
 - 30 g to 200 g/l of 1,2-propanediol;
 - 30 g to 150 g/l of vinyl alcohol resins;
 - 40 g to 600 g/l of water.
- 2. Concentrated aqueous emulsions according to claim 1, wherein said pyrethrinoid is deltamethrine, permethrine, cypermethrine, alphamethrine, tralomethrine, cyhalothrine, fenvalerate, cyfluthrine, flucythrinate, fluvalinate, fenpropathrine, tefluthrine and bifenthrine.
- 4. Preparation process for concentrated aqueous emulsions according to any one of claims 1 to 3, characterized in that:
- a solution A is prepared by dissolving an active ingredient in a solvent or solvents and a surface active agent is added to it;
- a solution B is prepared containing a film-forming agent, water and a co-

(11) AU-B-51697/90 (10) 629914

surface active agent

then solutions A and B are mixed together under agitation in order to obtain the desired aqueous emulsion.

5. Preparation process according to claim 4, wherein said surface active agent is selected from the group of acid phosphate ethers of formula:



in which R' represents a hydrogen atom, or represents an R(OCH₂CH₂)_n radical, R representing an alkyl radical containing up to 18 carbon atoms, an aryl radical containing up to 14 carbon atoms, or an alkylaryl radical containing up to 24 carbon atoms and n representing an integer which can vary between 1 to 18.

6. Preparation process according to claim 3 or claim 4, wherein said film-forming agent is a thermoplastic vinyl resin.

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Publiče

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(54) Title: NEW CONCENTRATED AQUEOUS EMULSIONS, PROCESS FOR PREPARING THEM AND THEIR USE IN THE FIELD OF PESTICIDES

(54) Titre: NOUVELLES EMULSIONS AQUEUSES CONCENTREES, LEUR PROCEDE DE PREPARATION ET LEUR APPLICATION DANS LE DOMAINE PESTICIDE

(57) Abstract

Provence (FR).

Concentrated aqueous emulsions, characterized in that they contain, in addition to the usual constituents of aqueous emulsions and the active principle, a film-forming agent and a surface-active agent chosen from the group of acid phosphate ethers. The emulsions are used, in particular, in the field of pesticides.

(57) Abrégé

L'invention a pour objet des émulsions aqueuses concentrées caractérisées en ce qu'elles renferment en plus des constituants habituels des émulsions aqueuses et du principe actif, un agent filmogène et un agent tensio-actif choisi dans le groupe des éthers phosphates acides. Les émulsions de l'invention trouvent notamment leur application dans le domaine des pesticides.

New concentrated aqueous emulsions, process for preparing them and their use in the field of pesticides.

The present invention relates to new concentrated aqueous emulsions, their preparation process and their use in the field of pesticides.

A subject of the invention is concentrated aqueous emulsions comprising:

- 0.01 g to 250 g/l of pyrethrinoid;
- g to 350 g/l of aromatic hydrocarbons;
- g to 350 g/l of phthalic ester;
- 40 g to 200 g/l of acid phosphate ether;
- 30 g to 200 g/l of 1,2-propanediol;
- 30 g to 150 g/l of vinyl alcohol resins;
- 40 g to 600 g/l of water.

The concentrated aqueous emulsions of the invention find their use in numerous fields, notably the field of pesticides. They offer an immediate biological effect and a good duration of action, it is this immediate and persistent double action which is the essential characteristic of the emulsions of the invention and allows their use in the agricultural field where they can form a protective film over vegetation after the evaporation of the dilution water.

Also a subject of the invention is preparation process for concentrated aqueous emulsions characterized in that:

- a solution A is prepared by dissolving the active ingredient in a solvent or solvents and the surface active agent is added to it;
- a solution B is prepared containing the film-forming agent, water and the co-surface active agent

then solutions A and B are mixed together under agitation in order to obtain the desired aqueous emulsion.

More particularly a subject of the invention is the film-forming agent which can be a thermoplastic vinyl resin.

Thermoplastic vinyl resins are distinguished by their solubility in water and their insolubility in virtually all other organic solvents.

In addition, these resins perform simultaneously the dual function of thickening and stabilising agent and of film-forming agent.

Among the thermoplastic resins preferred, there can be cited the resins belonging to the family of polyvinyl alcohols or also the resins derived from polyvinylpyrrolidone. These resins perform simultaneously the dual function of film-forming agent and of stabilizing, thickening agent.

Quite especially a subject of the invention is the surface active agent which can be a phosphoric ester of formula:

in which R' represents a hydrogen atom, or it represents a R(OCH₂CH₂)_n radical, R representing an alkyl radical containing up to 18 carbon atoms, an aryl radical containing up to 14 carbon atoms, or an alkylaryl radical containing up to 24 carbon atoms and n representing an integer which can vary from 1 to 18.

As an example of the preferred R radical, there can be cited the

$$C_9H_{19}$$
 radical.

In a preferred embodiment the pair of solvents must show the following characteristics:

- to dissolve the active ingredient of course;
- not to be miscible with water;
- not to be too volatile so as to contribute to the persistence of the active ingredient;
- to avoid crystallization of the active ingredient during storage for long periods at low temperatures;
- to have a density in the region of 1.

The pair of solvents can thus be constituted on the one hand by an aromatic hydrocarbon such as for example "SOLVESSO 150", and on the other hand by a derivative of phthalic acid namely, a mono or diester of phthalic acid

and of formula:

in which R and R', identical or different, represent an alkyl radical containing up to 8 carbon atoms, for example a methyl, ethyl, n-propyl, isopropyl or n-butyl radical.

The co-solvent is preferably a diol such as for example 1,2-propane-diol.

In a preferred embodiment the concentrated aqueous emulsions prepared contain a pesticide.

Among the preferred pesticides, there can be cited the pyrethrinoids such as deltamethrine, permethrine, cypermethrine, alphamethrine, tralomethrine, cyhalothrine, fenvalerate, cyfluthrine, flucythrinate, fluvalinate, fenpropathrine, tefluthrine and bifenthrine.

Also there can be cited the pyrethrinoids described and claimed in the European Patents 0038271, 0041021, 0048186, 0050534, 0110769, 01141012, 281439 and the French Patent 253639.

As an active ingredient the following can be considered suitable (1R,cis)-3-[(E)-2-fluoro-3-methoxy-3-oxo-1-propenyl]-2,2-dimethyl cyclopropanecarboxylate of 1,2,3,4,5-pentafluoro benzyl; or

(1R,cis)-2,2-dimethyl-3-[(Z)-3-oxo-3-[2,2,2-trifluoro-1-(trifluoromethyl)-ethoxy]-1-propenyl] cyclopropanecarboxylate of (S)-alpha-cyano-3-phenoxy benzyl.

desired aqueous emulsion.

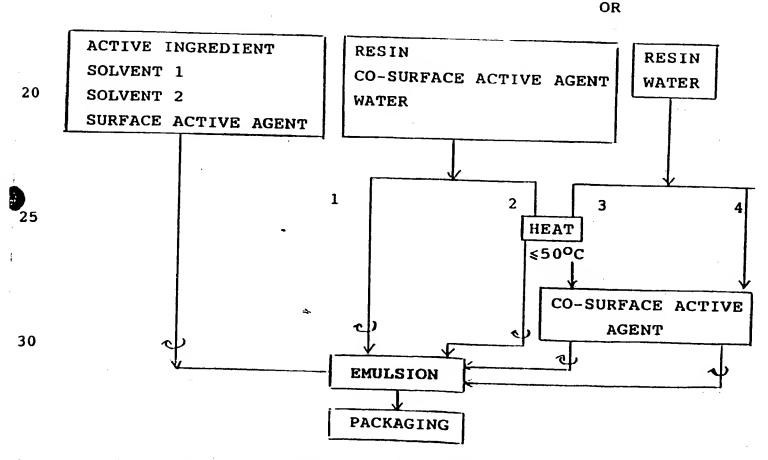
It is possible to add the following products:

- an anti-foaming agent, wetting products, anti-UV products;
- and/or bactericide products
- in liquid or powder form in the aqueous or organic phase (depending on their solubility) during the preparation.

The following examples illustrate the invention without however limiting it.

Examples 1 to 6

- The concentrated aqueous emulsions are made using as active ingredient:
 - (1R,cis)-2,2-dimethyl-3-[(Z)-3-oxo-3-[2,2,2-trifluoro-1-(trifluoromethyl)-ethoxy]-1-propenyl] cyclopropanecarboxylate of (S)-alpha-cyano-3-phenoxy benzyl hereafter called product
- 15 A, in accordance with the following diagram:



- 35 (1) Dissolving the polymer in the aqueous phase takes much longer.
 - (2) (3) After the "heating" operation a readjustment to ambient temperature of the aqueous phase is desirable before

proceeding with mixing.

(4) The solution probably shows the greatest speed of dissolution without recourse to a heat treatment.

The resume of the details of the constituents of the various emulsions prepared is given in the following table

		1					_
• •		Con	centrate	ed - aqı	eous -	emulsio	on
to.			(concer	ntration	is in g	/1.)	÷
•	Ingredients of formulation	No. 1	No.2	No.3	No.4	No.5	No.6
	PRODUCT A	100	150	150	150	150	150
15	SOLVESSO 150	52.5	100	100	100	100	100
	METHYL PHTHALATE	127.5	0	0	0	0	0
	ETHYL PHTHALATE	0	100	100	100	100	100
	AGRILAN F 546	120	0	0	0	0	o
	EMPHOS CS 136	0	70	0	0	0	0
20	BEYCOSTAT QA	0	0	70	0	60	o
	ESGANE 6236 A	0	0	0	70	0	60
	1,2-PROPANEDIOL	110	108.5	108.5	108.5	104	104
	MOWIOL 4/88	90	0	0	0	0	0
	MOWIOL 18/88	0	0	0	o	48	48
25	LUVISKOL K 90	- 0	41.5	41.5	41.5	0	0
	WATER	450	480	480	480	488	488

SOLVESSO 150

aromatic hydrocarbon

30

35

AGRILAN F 546 EMPHOS CS 136 BEYCOSTAT QA ESGANE 6236A

"Acid phosphate R-ether" type surface active agents

MOWIOL 4/88

MOWIOL 18/88

Polyvinyl alcohols with a different molecular weight but with the same saponification number.

LUVISKOL K 90

Polyvinyl pyrrolidone with a K number between 88 and 96

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Biological test:

The emulsions of the invention have been compared with a standard formulation of a concentrated emulsifiable type.

The tests have been carried out on apple tree acarida : Panonychus Ulmi.

The formulations of the invention were shown to be superior in terms of impact and persistence of action.

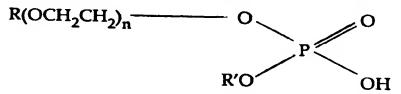
After 20 days, the formulations of the invention are still effective unlike the standard formulations.

The claims defining the invention are as follows:

- 1. Concentrated aqueous emulsions comprising:
 - 0.01 g to 250 g/l of pyrethrinoid;
 - g to 350 g/l of aromatic hydrocarbons;
 - 0 g to 350 g/l of phthalic ester;
 - 40 g to 200 g/l of acid phosphate ether;
 - 30 g to 200 g/l of 1,2-propanediol;
 - 30 g to 150 g/l of vinyl alcohol resins;
 - 40 g to 600 g/l of water.
- 2. Concentrated aqueous emulsions according to claim 1, wherein said pyrethrinoid is deltamethrine, permethrine, cypermethrine, alphamethrine, tralomethrine, cyhalothrine, fenvalerate, cyfluthrine, flucythrinate, fluvalinate, fenpropathrine, tefluthrine and bifenthrine.
- 3. Concentrated aqueous emulsions according to claim 1 or claim 2, substantially as herein described with reference to any one of the Examples.
- 4. Preparation process for concentrated aqueous emulsions according to any one of claims 1 to 3, characterized in that:
- a solution A is prepared by dissolving an active ingredient in a solvent or solvents and a surface active agent is added to it;
- a solution B is prepared containing a film-forming agent, water and a cosurface active agent

then solutions A and B are mixed together under agitation in order to obtain the desired aqueous emulsion.

5. Preparation process according to claim 4, wherein said surface active agent is selected from the group of acid phosphate ethers of formula:



in which R' represents a hydrogen atom, or represents an R(OCH2CH2)n radical,



R representing an alkyl radical containing up to 18 carbon atoms, an aryl radical containing up to 14 carbon atoms, or an alkylaryl radical containing up to 24 carbon atoms and n representing an integer which can vary between 1 to 18.

- 6. Preparation process according to claim 3 or claim 4, wherein said film-forming agent is a thermoplastic vinyl resin.
- 7. Preparation process according to claim 6, wherein said thermoplastic vinyl resin belongs to the family of polyvinyl alcohols.
- 8. Preparation process according to claim 6, wherein said thermoplastic vinyl resin is a derivative of polyvinylpyrrolidone.
- 9. Preparation process according to any one of claims 4 to 8, wherein said active ingredient is a pesticide.
- 10. Preparation process according to claim 9, wherein said pesticide is a pyrethrinoid.
- 11. Preparation process according to claim 10, wherein said pyrethrinoid is deltamethrine, permethrine, cypermethrine, alphamethrine, tralomethrine, cyhalothrine, fenvalerate, cyfluthrine, flucythrinate, fluvalinate, fenpropathrine, tefluthrine and bifenthrine.
- 12. Preparation process according to any one of claims 4 to 12, wherein said active ingredient, (1R,cis)-3-[(E)-2-fluoro-3-methoxy-3-oxo-1-propenyl]-2, 2-dimethyl cyclopropanecarboxylate of 1,2,3,4,5-pentafluoro benzyl; or (1R,cis)-2,2-dimethyl-3-[(Z)-3-oxo-3-[2,2,2,-trifluoro-1-(trifluoromethyl)-ethoxy]-1-propenyl] cyclopropanecarboxylate of (S)-alpha-cyano-3-phenoxy benzyl.
- 13. Preparation process for concentrated aqueous emulsions according to any one of claims 4 to 12, which process is substantially as herein described with reference to any one of the Examples.
- 14. Concentrated aqueous emulsions whenever prepared by the process as



claimed in any one of claims 4 to 13.

DATED this 30th day of April 1992.

ROUSSEL-UCLAF

By their Patent Attorneys:

CALLINAN LAWRIE

Michael J. Hordihan.



	•	International Application No PC	T/FR 90/00109
I. CLASS	FICATION OF SUBJECT MATTER (If several classif	ication symbols epply, Indicete ell) *	
	to International Patent Classification (IPC) or to both Netto	onal Classification and IPC	
Int.	Cl. ⁵ A O1 N 25/30, A O1 N 25/2	24	
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III. DOCU	MENTS CONSIDERED TO BE RELEVANT		
ategory •	Citation of Document, 13 with indication, where appr	opriale, of the relevant pessages 12	Relevant to Claim No. 13
X	EP, A, 0118759 (HOECHST AG) 19 see page 5, line 14 - page lines 27-29; claims 1-4,8		1-5,7
X	EP, A, 0224846 (HOECHST AG) 10 see page 4, lines 6-12,35- 1-3; claims 1,2,4-6	1-4,7-9	
Α	EP, A, 0190995 (CIBA-GEIGY) 13 see page 6, paragraph 3; c		1-12
A	EP, A, 0022666 (SAMPSON) 21 Ja see page 1, lines 15-20; page 4, lines 15-20; page claims 1,3,5	page 2, lines 3-8;	1-12
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"A" doc con "E" earl filin "L" doc cita "O" doc oth "P" doc late	categories of cited documents: 10 ument defining the general state of the art which is not pldered to be of particular relevance or document but published on or after the international grade grade ument which may throw doubts on priority claim(s) or the is cited to establish the publication date of another ion or other special reason (as specified) ument referring to an oral disclosure, use, exhibition or or means ument published prior to the international filing date but than the priority date claimed	"T" later document published efter of priority date end not in conficited to understand the princip invention "X" document of particular relevant cannot be considered novel or involve an inventive step "Y" document of particular relevant cannot be considered to involve document is combined with one ments, such combination being in the art. "A" document member of the same	lict with the application but le or theory underlying the ace; the cleimed invention r cannot be considered to ace; the claimed invention an inventive step when the e or more other such docu- obvious to a person skilled
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	y 1990 (23.05.90)	21 June 1990 (21.06.	90)
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III. DOCUM	HENTS CONSIDERED TO SE RELEVANT (CONTINUED FROM THE SECOND SHE	m
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A	EP, A, 0197293 (BAYER AG) 15 October 1986 see claims	1-12
A	FR. A, 2599220 (SOCIETE MEDITERRANEENNE D'AEROSOLS et al.) 4 December 1987, see page 2, line 10 - page 3, line 15; page 4, lines 4,6,16-19; page 6, example 2	1-12
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 15/06/90 The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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IV. CERTI	FICATION		
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	ion chargée de la recherche internationale	Signature du toncironpeire autorisé	XX
OF	FICE EUROPEEN DES BREVETS		

III. DOCUI	NTS CONSIDÉRÉS COMME PERTINENTS (SUITE DES RENSEIGNEMENTS INDIQUÉS SUR LA DEUXIÈME FEUILLE)		
Catégorie *	identification des documents cités, avec indication, ai nécessaire, des passages pertinents	Nº des revendications visées	
Ā	EP, A, 0022666 (SAMPSON) 21 janvier 1981 voir page 1, lignes 15-20; page 2, lignes 3-8; page 4, lignes 15-20; page 5, lignes 21-23; revendications 1,3,5	1-12	
A	EP, A, 0197293 (BAYER AG) 15 octobre 1986 voir revendications	1-12	
A	FR, A, 2599220 (SOCIETE MEDITERRANEENNE D'AEROSOLS et al.) 4 décembre 1987 voir page 2, ligne 10 - page 3, ligne 15; page 4, lignes 4,6,16-19; page 6, exemple 2	1-12	
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ANNEXE AU RAPPORT DE RECHERCHE INTERNATIONALE RELATIF A LA DEMANDE INTERNATIONALE NO.

FR 9000109 SA 35004

La présente annexe indique les membres de la famille de brevets relatifs aux documents brevets cités dans le rapport de recherche internationale visé ci-dessus.

Lesdits membres sont contenus au fichier informatique de l'Office européen des brevets à la date du 15/06/90 Les renseignements fournis sont donnés à titre indicatif et n'engagent pas la responsabilité de l'Office européen des brevets.

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